

## **Analytical Laboratory**

13339 Hagers Ferry Road Huntersville, NC 28078-7929 McGuire Nuclear Complex - MG03A2 Phone: 980-875-5245 Fax: 980-875-4349

### **Order Summary Report**

Order Number:	J11110004							
Customer Name(s):	Bill Kennedy, Melonie Martin, Wayne	Chapman,	Tom Johnson					
Customer Address:	3195 Pine Hall Rd							
	Mailcode: Belews Steam Station							
	Belews Creek, NC 28012							
Lab Contact:	Jason C Perkins	Phone:	980-875-5348					
Report Authorized By: (Signature)		Date	<b>)</b> :	11/22/2011				

### **Program Comments:**

Please contact the Program Manager (Jason C Perkins) with any questions regarding this report.

#### **Data Flags & Calculations:**

Any analytical tests or individual analytes within a test flagged with a Qualifier indicate a deviation from the method quality system or quality control requirement. The qualifier description is found at the end of the Certificate of Analysis (sample results) under the qualifiers heading. All results are reported on a dry weight basis unless otherwise noted.

### Data Package:

This data package includes analytical results that are applicable only to the samples described in this narrative. An estimation of the uncertainty of measurement for the results in the report is available upon request. This report shall not be reproduced, except in full, without the written consent of the Analytical Laboratory. Please contact the Analytical laboratory with any questions. The order of individual sections within this report is as follows:

Job Summary Report, Sample Identification, Technical Validation of Data Package, Analytical Laboratory Certificate of Analysis, Analytical Laboratory QC Reports, Sub-contracted Laboratory Results, Customer Specific Data Sheets, Reports & Documentation, Customer Database Entries, Test Case Narratives, Chain of Custody (COC)

#### Certification:

The Analytical Laboratory holds the following State Certifications: North Carolina (DENR) Certificate #248, South Carolina (DHEC) Laboratory ID # 99005. Contact the Analytical Laboratory for definitive information about the certification status of specific methods.

Sample ID	Plant/Station	Collection Date and Time	Collected By	Sample Description
2011023685	BELEWS	09-Nov-11 7:30 AM	W. B. WORKMAN	FGD Purge Eff
2011023686	BELEWS	09-Nov-11 7:35 AM	W. B. WORKMAN	EQ TANK EFF.
2011023687	BELEWS	09-Nov-11 7:40 AM	W. B. WORKMAN	BIOREACTOR 1 INF.
2011023688	BELEWS	09-Nov-11 7:45 AM	W. B. WORKMAN	BIOREACTOR 2 INF.
2011023689	BELEWS	09-Nov-11 7:50 AM	W. B. WORKMAN	BIOREACTOR 2 EFF.
2011023692	BELEWS	09-Nov-11 7:55 AM	W. B. WORKMAN	FILTER BLANK
2011023693	BELEWS	09-Nov-11 8:00 AM	W. B. WORKMAN	Trip Blank
2011023694	BELEWS	09-Nov-11 1:10 PM	DAVID MORRIS	BIOREACTOR 1 INF.
2011023695	BELEWS	09-Nov-11 1:10 PM	DAVID MORRIS	HG BLANK BIOREACTOR 1 INF.
2011023696	BELEWS	09-Nov-11 1:20 PM	DAVID MORRIS	BIOREACTOR 2 INF.
2011023697	BELEWS	09-Nov-11 1:20 PM	DAVID MORRIS	Hg Blk BioReactor 2 Inf
2011023698	BELEWS	09-Nov-11 1:15 PM	DAVID MORRIS	BIOREACTOR 2 EFF.
2011023699	BELEWS	09-Nov-11 1:15 PM	DAVID MORRIS	Hg Blk BioReactor 2 Eff

### **Checklist:**

Reviewed By:

DataBase Administrator

	COC and .pdf report are in agreement with sample and analyses (compliance programs and procedure		<b>✓</b> Yes	No
	All Results are less than the laboratory reporting lim	its.	Yes	<b>✓</b> No
	All laboratory QA/QC requirements are acceptable.	<b>✓</b> Yes	☐ No	
	The Vendor Laboratories have been qualified by the Analytical Laboratory	<b>;</b>	Yes	
Report S	ections Included:			
<b>✓</b> Jol	b Summary Report	✓ Sub-contr	acted Laborate	ory Results
<b>✓</b> Sa	mple Identification	☐ Customer	Specific Data	Sheets, Reports, & Documentation
<b>✓</b> Te	chnical Validation of Data Package	☐ Customer	Database Ent	ries
<b>✓</b> An	alytical Laboratory Certificate of Analysis	✓ Chain of C	Custody	
☐ An	alytical Laboratory QC Report	<b>✓</b> Electronic	: Data Delivera	able (EDD) Sent Separately

Date:

11/22/2011

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### Order # J11110004

Site: FGD Purge Eff Sample #: 2011023685

Collection Date: 09-Nov-11 7:30 AM Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	Method	Analysis Date/Time	Analyst
INORGANIC IONS BY IC							
Bromide	94	mg/L		5	EPA 300.0	15-Nov-11 00:21	JAHERMA
MERCURY (COLD VAPOR) IN	WATER						
Mercury (Hg)	247	ug/L		5	EPA 245.1	18-Nov-11 08:44	AGIBBS
TOTAL RECOVERABLE META	ALS BY ICP						
Boron (B)	158	mg/L		0.5	EPA 200.7	15-Nov-11 14:39	DJSULL1
DISSOLVED METALS BY ICP-	-MS						
Selenium (Se)	267	ug/L		10	EPA 200.8	14-Nov-11 11:47	KRICHAR
TOTAL RECOVERABLE META	ALS BY ICP-MS						
Arsenic (As)	164	ug/L		10	EPA 200.8	15-Nov-11 11:55	KRICHAR
Chromium (Cr)	191	ug/L		10	EPA 200.8	15-Nov-11 11:55	KRICHAR
Copper (Cu)	106	ug/L		10	EPA 200.8	15-Nov-11 11:55	KRICHAR
Nickel (Ni)	172	ug/L		10	EPA 200.8	15-Nov-11 11:55	KRICHAR
Selenium (Se)	4220	ug/L		10	EPA 200.8	15-Nov-11 11:55	KRICHAR
Silver (Ag)	< 10	ug/L		10	EPA 200.8	15-Nov-11 11:55	KRICHAR
Zinc (Zn)	203	ug/L		20	EPA 200.8	15-Nov-11 11:55	KRICHAR
SELENIUM SPECIATION							
Vendor Parameter	Complet	te			V_AS&C		
TOTAL DISSOLVED SOLIDS							
TDS	17000	mg/L		200	SM2540C	15-Nov-11 14:50	TJA7067

Site: EQ TANK EFF. Sample #: 2011023686

Collection Date: 09-Nov-11 7:35 AM Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	Method	Analysis Date/Time	Analyst		
MERCURY (COLD VAPOR) IN WA	<u>TER</u>								
Mercury (Hg)	173	ug/L		2.5	EPA 245.1	18-Nov-11 08:46	AGIBBS		
TOTAL RECOVERABLE METALS BY ICP									
Boron (B)	156	mg/L		0.5	EPA 200.7	15-Nov-11 14:43	DJSULL1		
DISSOLVED METALS BY ICP-MS									
Selenium (Se)	195	ug/L		10	EPA 200.8	14-Nov-11 11:51	KRICHAR		

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### Order # J11110004

Site: EQ TANK EFF. Sample #: 2011023686

Collection Date: 09-Nov-11 7:35 AM Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	Method	Analysis Date/Time	Analyst
TOTAL RECOVERABLE METALS E	BY ICP-MS						
Arsenic (As)	137	ug/L		10	EPA 200.8	15-Nov-11 11:58	KRICHAR
Chromium (Cr)	155	ug/L		10	EPA 200.8	15-Nov-11 11:58	KRICHAR
Copper (Cu)	87.6	ug/L		10	EPA 200.8	15-Nov-11 11:58	KRICHAR
Nickel (Ni)	157	ug/L		10	EPA 200.8	15-Nov-11 11:58	KRICHAR
Selenium (Se)	3690	ug/L		10	EPA 200.8	15-Nov-11 11:58	KRICHAR
Silver (Ag)	< 10	ug/L		10	EPA 200.8	15-Nov-11 11:58	KRICHAR
Zinc (Zn)	177	ug/L		20	EPA 200.8	15-Nov-11 11:58	KRICHAR

Site: BIOREACTOR 1 INF. Sample #: 2011023687

Collection Date: 09-Nov-11 7:40 AM Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	Method	Analysis Date/Time	Analyst				
TOTAL RECOVERABLE METALS B	Y ICP										
Boron (B)	151	mg/L		0.5	EPA 200.7	15-Nov-11 14:47	DJSULL1				
DISSOLVED METALS BY ICP-MS											
Selenium (Se)	137	ug/L		10	EPA 200.8	14-Nov-11 11:54	KRICHAR				
TOTAL RECOVERABLE METALS BY ICP-MS											
Arsenic (As)	< 10	ug/L		10	EPA 200.8	15-Nov-11 12:01	KRICHAR				
Chromium (Cr)	< 10	ug/L		10	EPA 200.8	15-Nov-11 12:01	KRICHAR				
Copper (Cu)	< 10	ug/L		10	EPA 200.8	15-Nov-11 12:01	KRICHAR				
Nickel (Ni)	57.9	ug/L		10	EPA 200.8	15-Nov-11 12:01	KRICHAR				
Selenium (Se)	130	ug/L		10	EPA 200.8	15-Nov-11 12:01	KRICHAR				
Silver (Ag)	< 10	ug/L		10	EPA 200.8	15-Nov-11 12:01	KRICHAR				
Zinc (Zn)	< 20	ug/L		20	EPA 200.8	15-Nov-11 12:01	KRICHAR				
SELENIUM SPECIATION											
Vendor Parameter	Complete	)			V_AS&C						

Site: BIOREACTOR 2 INF. Sample #: 2011023688

Collection Date: 09-Nov-11 7:45 AM Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	Method	Analysis Date/Time	Analyst
TOTAL RECOVERABLE METALS	BY ICP						
Boron (B)	153	mg/L		0.5	EPA 200.7	15-Nov-11 14:51	DJSULL1

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### Order # J11110004

Site: BIOREACTOR 2 INF.

Collection Date: 09-Nov-11 7:45 AM

Sample #: 2011023688

Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	Method	Analysis Date/Time	Analyst
TOTAL RECOVERABLE METALS	BY ICP-MS						
Arsenic (As)	< 10	ug/L		10	EPA 200.8	15-Nov-11 12:05	KRICHAR
Chromium (Cr)	< 10	ug/L		10	EPA 200.8	15-Nov-11 12:05	KRICHAR
Copper (Cu)	< 10	ug/L		10	EPA 200.8	15-Nov-11 12:05	KRICHAR
Nickel (Ni)	< 10	ug/L		10	EPA 200.8	15-Nov-11 12:05	KRICHAR
Selenium (Se)	14.6	ug/L		10	EPA 200.8	15-Nov-11 12:05	KRICHAR
Silver (Ag)	< 10	ug/L		10	EPA 200.8	15-Nov-11 12:05	KRICHAR
Zinc (Zn)	< 20	ug/L		20	EPA 200.8	15-Nov-11 12:05	KRICHAR

Site: BIOREACTOR 2 EFF. Sample #: 2011023689

Collection Date: 09-Nov-11 7:50 AM Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	Method	Analysis Date/Time	Analyst				
INORGANIC IONS BY IC											
Bromide	88	mg/L		5	EPA 300.0	15-Nov-11 00:37	JAHERMA				
MERCURY (COLD VAPOR) IN WAT	ER										
Mercury (Hg)	<1	ug/L		1	EPA 245.1	18-Nov-11 09:03	AGIBBS				
TOTAL RECOVERABLE METALS BY ICP											
Boron (B)	166	mg/L		0.5	EPA 200.7	15-Nov-11 14:55	DJSULL1				
TOTAL RECOVERABLE METALS BY ICP-MS											
Arsenic (As)	< 5	ug/L		5	EPA 200.8	15-Nov-11 12:08	KRICHAR				
Chromium (Cr)	< 5	ug/L		5	EPA 200.8	15-Nov-11 12:08	KRICHAR				
Copper (Cu)	< 5	ug/L		5	EPA 200.8	15-Nov-11 12:08	KRICHAR				
Nickel (Ni)	< 5	ug/L		5	EPA 200.8	15-Nov-11 12:08	KRICHAR				
Selenium (Se)	< 5	ug/L		5	EPA 200.8	15-Nov-11 12:08	KRICHAR				
Silver (Ag)	< 5	ug/L		5	EPA 200.8	15-Nov-11 12:08	KRICHAR				
Zinc (Zn)	< 10	ug/L		10	EPA 200.8	15-Nov-11 12:08	KRICHAR				
SELENIUM SPECIATION											
Vendor Parameter	Complete	)			V_AS&C						

Site: FILTER BLANK Sample #: 2011023692

Collection Date: 09-Nov-11 7:55 AM Matrix: OTHER

Analyte	Result	Units	Qualifiers	RDL	Method	Analysis Date/Time	Analyst
DISSOLVED METALS BY ICP-MS							
Selenium (Se)	1.10	ug/L		1	EPA 200.8	14-Nov-11 11:31	KRICHAR

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### Order # J11110004

Site: Trip Blank Sample #: 2011023693 OTHER Collection Date: 09-Nov-11 8:00 AM Matrix: Analyte Result Units Qualifiers **RDL** Method **Analysis Date/Time** Analyst **TOTAL RECOVERABLE METALS BY ICP** Boron (B) 0.05 DJSULL1 < 0.05 mg/L EPA 200.7 15-Nov-11 14:23 **TOTAL RECOVERABLE METALS BY ICP-MS KRICHAR** Arsenic (As) < 1 ug/L 1 EPA 200.8 15-Nov-11 11:43 Chromium (Cr) < 1 ug/L 1 EPA 200.8 15-Nov-11 11:43 **KRICHAR** Copper (Cu) ug/L EPA 200.8 15-Nov-11 11:43 **KRICHAR** Nickel (Ni) < 1 ug/L 1 EPA 200.8 15-Nov-11 11:43 **KRICHAR** Selenium (Se) < 1 ug/L 1 **EPA 200.8** 15-Nov-11 11:43 **KRICHAR** Silver (Ag) < 1 ug/L EPA 200.8 15-Nov-11 11:43 **KRICHAR** < 2 ug/L 2 EPA 200.8 15-Nov-11 11:43 **KRICHAR** Zinc (Zn) **SELENIUM SPECIATION** Vendor Parameter Complete V\_AS&C Site: BIOREACTOR 1 INF. Sample #: 2011023694 Collection Date: 09-Nov-11 1:10 PM Matrix: OTHER Qualifiers RDL Method Analyte Result Units Analysis Date/Time **Analyst MERCURY 1631** Vendor Parameter Complete V BRAND Site: HG BLANK BIOREACTOR 1 INF. Sample #: 2011023695 Collection Date: 09-Nov-11 1:10 PM Matrix: OTHER Analyte Result Units Qualifiers **RDL** Method **Analysis Date/Time Analyst MERCURY 1631** Vendor Parameter V BRAND Complete Site: BIOREACTOR 2 INF. Sample #: 2011023696 Collection Date: 09-Nov-11 1:20 PM Matrix: OTHER Analyte Result Units Qualifiers **RDL** Method Analysis Date/Time Analyst MERCURY 1631 Vendor Parameter Complete V BRAND

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### Order # J11110004

Site: Hg Blk BioReactor 2 Inf

Collection Date: 09-Nov-11 1:20 PM

Sample #: 2011023697

Matrix: OTHER

Analyte Result Units Qualifiers RDL Method Analysis Date/Time Analyst

MERCURY 1631

Vendor Parameter Complete V\_BRAND

Site: BIOREACTOR 2 EFF. Sample #: 2011023698

Collection Date: 09-Nov-11 1:15 PM Matrix: OTHER

Analyte Result Units Qualifiers RDL Method Analysis Date/Time Analyst

MERCURY 1631

Vendor Parameter Complete V\_BRAND

Site: Hg Blk BioReactor 2 Eff Sample #: 2011023699

Collection Date: 09-Nov-11 1:15 PM Matrix: OTHER

Analyte Result Units Qualifiers RDL Method Analysis Date/Time Analyst

MERCURY 1631

Vendor Parameter Complete V\_BRAND



18804 Northcreek Parkway Bothell, WA, 98011 Tel: (425) 483-3300 Fax: (425) 483-9818 www.appliedspeciation.com

November 14, 2011

Jay Perkins Duke Energy Analytical Laboratory Mail Code MGO3A2 (Building 7405) 13339 Hagers Ferry Rd. Huntersville, NC 28078 (704) 875-5245

Project: Belews – FGD WWTS Bi-Monthly Sampling) (LIMS # J11110004)

Dear Mr. Perkins,

Attached is the report associated with four (4) aqueous samples submitted for selenium speciation analysis on November 10, 2011. The samples were received in a sealed cooler at -0.3°C on November 11, 2011. Selenium speciation analysis was performed via ion chromatography inductively coupled plasma dynamic reaction cell mass spectrometry (IC-ICP-DRC-MS). Any issues associated with the analysis are addressed in the following report.

If you have any questions, please feel free to contact me at your convenience.

Sincerely,

Russell Gerads Vice President

Applied Speciation and Consulting, LLC

### Applied Speciation and Consulting, LLC

Report prepared for:

Jay Perkins Duke Energy Analytical Laboratory Mail Code MGO3A2 (Building 7405) 13339 Hagers Ferry Rd. Huntersville, NC 28078

Project: Belews – FGD WWTS Bi-Monthly Sampling) (LIMS # J11110004)

November 14, 2011

### 1. Sample Reception

Four (4) aqueous samples in 125mL HDPE bottles (provided by Applied Speciation and Consulting) were submitted for selenium speciation analysis on November 10, 2011. The samples were received on November 11, 2011 in a sealed container at -0.3°C.

The samples were received in a laminar flow clean hood, void of trace metals contamination and ultra-violet radiation, and designated a discrete sample identifier. An aliquot of each sample was filtered (0.45µm) and each filtrate was stored in a secure, monitored cryofreezer (maintained at a temperature of -80°C) until selenium speciation analysis could be performed via ion chromatography inductively coupled plasma dynamic reaction cell mass spectrometry (IC-ICP-DRC-MS).

### 2. Sample Preparation

All sample preparation is performed in laminar flow clean hoods known to be free from trace metals contamination. All applied water for dilutions and sample preservatives are monitored for contamination to account for any biases associated with the sample results.

<u>Selenium Speciation Analysis by IC-ICP-DRC-MS</u> Prior to analysis, an aliquot of each sample was filtered with a syringe filter (0.45µm) and injected directly into a sealed autosampler vial. No further sample preparation was performed as any chemical alteration of a sample may shift the equilibrium of the system, resulting in changes in speciation ratios.

### 3. Sample Analysis

All sample analysis is preceded by a minimum of a five-point calibration curve spanning the entire concentration range of interest. Calibration curves are performed at the beginning of

each analytical day. All calibration curves, associated with each species of interest, are standardized by linear regression resulting in a response factor. All sample results are **instrument blank corrected** to account for any operational biases associated with the analytical platform.

Prior to sample analysis, all calibration curves are verified using second source standards which are identified as initial calibration verification standards (ICV).

Ongoing instrument performance is identified by the analysis of continuing calibration verification standards (CCV) and continuing calibration blanks (CCB) at a minimal interval of every ten analytical runs.

Selenium Speciation Analysis by IC-ICP-DRC-MS Each sample for selenium speciation analysis was analyzed by ion chromatography inductively coupled plasma dynamic reaction cell mass spectrometry (IC-ICP-DRC-MS) on November 11, 2011. An aliquot of each sample is injected onto an anion exchange column and mobilized by a basic (pH > 7) gradient. The eluting selenium species are then introduced into a radio frequency (RF) plasma where energy-transfer processes cause desolvation, atomization, and ionization. The ions are extracted from the plasma through a differentially-pumped vacuum interface and travel through a pressurized chamber (DRC) containing a specific reactive gas which preferentially reacts with interfering ions of the same target mass to charge ratios (m/z). A solid-state detector detects ions transmitted through the mass analyzer and the resulting current is processed by a data handling system.

Retention times for each eluting species are compared to known standards for species identification.

### 4. Analytical Issues

The overall analyses went well and no significant analytical issues were encountered. All quality control parameters associated with this sample were within acceptance limits.

The estimated method detection limits (eMDLs) for selenite, selenate, and selenocyanate are generated from replicate analyses of the lowest standard in the calibration curve. Not all selenium species are present in preparation blanks; therefore, eMDL calculations based on preparation blanks are artificially biased low.

The eMDL for methylseleninic acid and selenomethionine is calculated from the average eMDL of selenite, selenate, and selenocyanate. The calibration does not contain methylseleninic acid or selenomethionine due to impurities in these standards which would bias the results for other selenium species.

All selenium speciation results have been corrected for instrument drift in accordance with the continuing calibration verification standards. All quality control parameters were within acceptance limits signifying that the applied correction was appropriate.

If you have any questions or concerns regarding this report, please feel free to contact me.

Sincerely,

Russell Gerads Vice President

Applied Speciation and Consulting, LLC

# Selenium Speciation Results for Duke Energy Project Name: Belews - FGD WWTS Bi-Monthly Sampling Contact: Jay Perkins LIMS #J11110004

Date: November 14, 2011 Report Generated by: Russell Gerads Applied Speciation and Consulting, LLC

### Sample Results

						Unknown Se
Sample ID	Se(IV)	Se(VI)	SeCN	MeSe(IV)	SeMe	Species (n)
FGD Purge Eff	176	68.9	ND (<4.3)	ND (<5.1)	ND (<5.1)	4.8 (1)
BioReactor 1 Inf	46.5	63.9	ND (<1.1)	4.7	ND (<1.3)	0 (0)
BioRector 2 Eff	ND (<1.2)	ND (<1.5)	ND (<1.1)	ND (<1.3)	ND (<1.3)	0 (0)
Metals Trip Blk	ND (<0.24)	ND (<0.30)	ND (<0.22)	ND (<0.25)	ND (<0.25)	0 (0)

All results reflect the applied dilution and are reported in µg/L

ND = Not detected at the applied dilution

SeCN = Selenocyanate

MeSe(IV) = Methylseleninic acid

SeMe = Selenomethionine

Unknown Se Species = Total concentration of all unknown Se species observed by IC-ICP-MS

n = number of unknown Se species observed

# Selenium Speciation Results for Duke Energy Project Name: Belews - FGD WWTS Bi-Monthly Sampling Contact: Jay Perkins LIMS #J11110004

Date: November 14, 2011 Report Generated by: Russell Gerads Applied Speciation and Consulting, LLC

### **Quality Control Summary - Preparation Blank Summary**

Analyte (μg/L)	PBW1	PBW2	PBW3	PBW4	Mean	StdDev	eMDL*	eMDL 10x	eMDL 50x	eMDL 200x
Se(IV)	0.000	0.000	0.000	0.000	0.000	0.000	0.024	0.24	1.2	4.8
Se(VI)	0.000	0.000	0.000	0.000	0.000	0.000	0.030	0.30	1.5	6.1
SeCN	0.000	0.000	0.000	0.000	0.000	0.000	0.022	0.22	1.1	4.3
MeSe(IV)	0.000	0.000	0.000	0.000	0.000	0.000	0.025	0.25	1.3	5.1
SeMe	0.000	0.000	0.000	0.000	0.000	0.000	0.025	0.25	1.3	5.1

eMDL = Estimated Method Detection Limit

### **Quality Control Summary - Certified Reference Materials**

Analyte (µg/L)	CRM	True Value	Result	Recovery
Se(IV)	LCS	9.57	9.76	102.0
Se(VI)	LCS	9.48	9.55	100.8
SeCN	LCS	8.92	9.43	105.8
MeSe(IV)	LCS	6.47	6.38	98.6
SeMe	LCS	9.32	9.64	103.5

<sup>\*</sup>Please see narrative regarding eMDL calculations

# Selenium Speciation Results for Duke Energy Project Name: Belews - FGD WWTS Bi-Monthly Sampling Contact: Jay Perkins LIMS #J11110004

Date: November 14, 2011 Report Generated by: Russell Gerads Applied Speciation and Consulting, LLC

### **Quality Control Summary - Matrix Duplicates**

Analyte (µg/L)	Sample ID	Rep 1	Rep 2	Mean	RPD
Se(IV)	FGD Purge Eff	176.2	166.6	171.4	5.6
Se(VI)	FGD Purge Eff	68.9	71.0	70.0	3.1
SeCN	FGD Purge Eff	ND (<4.3)	ND (<4.3)	NC	NC
MeSe(IV)	FGD Purge Eff	ND (<5.1)	ND (<5.1)	NC	NC
SeMe	FGD Purge Eff	ND (<5.1)	ND (<5.1)	NC	NC

ND = Not detected at the applied dilution

NC = Value was not calculated due to one or more concentrations below the eMDL

### **Quality Control Summary - Matrix Spike/ Matrix Spike Duplicate**

Analyte (µg/L)	Sample ID	Spike Conc	MS Result	Recovery	Spike Conc	MSD Result	Recovery	RPD
Se(IV)	FGD Purge Eff	1112	1325	103.7	1112	1358	106.7	2.5
Se(VI)	FGD Purge Eff	1009	1093	101.4	1009	1091	101.1	0.2
SeCN	FGD Purge Eff	915.0	868.8	95.0	915.0	878.4	96.0	1.1

Comments:	9)SeallLpcked By	7Railinguisness By	5)Redinguished By	a) Relimpulgities By	1) Reijnguished By				82	0	8		Ø ≫⊙ olumn:	•	0 0	70	3 × 1/1/2 13 08 1	that to	LAB USE ONLY		Cus 8)Oper, Unit:		est Comp 2) Client:		A)Project Name		DU	7.1.7.1.1.4.1.1.1
· B by ICP	tuis.	ic (i.g.)	ŝ	Jan	Charles to sale	4	siome	10.0	01110	gie A	349 (5)11							5	Se Speciation Bottle			(6)P	Bill Kennedy, Melonie Martin, Wayne Chapman, Tom Johnson	WWTS BI-Mon	Relews	ergy	ke	Z. J
As, Cr, Cu, Ni, Se, Ag, Zn by IMS	/// Date/Time	Dale/Time	Data/Time)	Date/Time		atenue to son & data halow - fill out from left to right.		,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,,	Metals Trio Bik	Filter Blk	BioReactor 2		BioReactor 2		BioReactor 1 Inf	EQ Tank Eff.	FGD Purge Eff	<sup>13</sup> Sample Description or ID			, J. P. C.		*	13	FGB	Huntersville, N. C. 280 (704) 875-5245 Fax: (704) 875-4349	Duke Energy Allalytica: Labora Mail Code MGO3A2 (Building 7405) 13339 Haders Ferry Rd	
	1300			· -	13/00/15			H	io Bik	¥	r 2 Eff		r 2 Inf		1 Inf	Eff.	e Eff	iption or ID				Mail Code:			e No:	28078 5 349	Allialytica: Laboratory  1GO3A2 (Building 7405)  Hagers Ferry Rd	- Moreron
Digestions = TRM	(0) Seall Lock Opened By	Accepted By	Accepted By:	Acceptant By	Accepted By M			_	11/9/11 8:00	11/9/11 7:55	080.07Em		11/9/11 7:45		119/117:40	11/9/11 7:35	1	Date Time		Sampling conducted; 2nd and 4th Wednesday	Customer to complete all appropriate non-shaded areas.		PO#133241	AS&C		ogged by Dai	THADOU	
thomas.d.john	R		<b>\</b>	Š.				Filtering				-					W. Working	Signature		nd and 4th Wednesday	-shaded areas.		2=H,SO, 4=lice	Cooler Temp (C)		11/10/11 083	MATRIX: OTHER Samples Originating From	Analytical Lab
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November 21, 2011

Duke Energy
ATTN: Jay Perkins
Scientific Support-Laboratory
13339 Hagers Ferry Road
Huntersville NC 28078
jcperkins@duke-energy.com
labcustomer@duke-energy.com

RE: Project DUK-HV1101 Client Project: J11110004

Dear Mr. Perkins,

On November 11, 2011, Brooks Rand Labs (BRL) received three (3) wastewater samples and three (3) corresponding field blanks. Samples were logged-in for total mercury (Hg) analysis. All samples were received, prepared, analyzed, and stored according to BRL SOPs and EPA methodology.

The results were blank-corrected as described in the calculations section of the applicable SOP(s) and may be evaluated using adjusted reporting limits to account for sample aliquot size. Please refer to the *Sample Results* page for sample-specific detection limits and other details.

BRL, an accredited laboratory, certifies the reported results of all analyses for which BRL is NELAP accredited meet all NELAP requirements. For more details, see the *Report Information* page of the report.

Please feel free to contact me if you have any questions regarding this report.

Sincerely,

Tiffany Stilwater Project Manager

tiffany@brooksrand.com

tilwate



Page 18 of 29 Client PM: Jay Perkins Client PO: 141391

## Report Information

### **Laboratory Accreditation**

BRL is accredited by the *National Environmental Laboratory Accreditation Program* (NELAP) through the State of Florida Department of Health, Bureau of Laboratories (E87982) and is certified to perform many environmental analyses. BRL is also certified by many other states to perform environmental analyses. For a current list of our accreditations/certifications, please visit our website at <a href="http://www.brooksrand.com/default.asp?contentID=586">http://www.brooksrand.com/default.asp?contentID=586</a>. Results reported relate only to the samples listed in the report.

### **Field Quality Control Samples**

Please be notified that certain EPA methods require the collection of field quality control samples of an appropriate type and frequency; failure to do so is considered a deviation from some methods and for compliance purposes should only be done with the approval of regulatory authorities. Please see the specific EPA methods for details regarding required field quality control samples.

#### **Common Abbreviations**

BLK	method blank	MS	matrix spike
BRL	Brooks Rand Labs	MSD	matrix spike duplicate
BS	laboratory fortified blank	ND	non-detect
CAL	calibration standard	NR	non-reportable
CCV	continuing calibration verification	PS	post preparation spike
COC	chain of custody record	REC	percent recovery
CRM	certified reference material	RPD	relative percent difference
D	dissolved fraction	RSD	relative standard deviation
DUP	duplicate	SCV	secondary calibration verification
ICV	initial calibration verification	SOP	standard operating procedure
MDL	method detection limit	SRM	standard reference material
MRL	method reporting limit	T	total recoverable fraction

### **Definition of Data Qualifiers**

(Effective 9/23/09)

- B Detected by the instrument, the result is > the MDL but ≤ the MRL. Result is reported and considered an estimate.
- **E** An estimated value due to the presence of interferences. A full explanation is presented in the narrative.
- **H** Holding time and/or preservation requirements not met. Result is estimated.
- **J** Estimated value. A full explanation is presented in the narrative.
- J-M Duplicate precision (RPD) for associated QC sample was not within acceptance criteria. Result is estimated.
- J-N Spike recovery for associated QC sample was not within acceptance criteria. Result is estimated.
- M Duplicate precision (RPD) was not within acceptance criteria. Result is estimated.
- N Spike recovery was not within acceptance criteria. Result is estimated.
- **R** Rejected, unusable value. A full explanation is presented in the narrative.
- U Result is ≤ the MDL or client requested reporting limit (CRRL). Result reported as the MDL or CRRL.
- X Result is not BLK-corrected and is within 10x the absolute value of the highest detectable BLK in the batch. Result is estimated.

These qualifiers are based on those previously utilized by Brooks Rand, Ltd., those found in the EPA <u>SOW ILM03.0</u>, Exhibit B, Section III, pg. B-18, and the <u>USEPA Laboratory Data Validation Functional Guidelines for Evaluating Inorganic Analyses; USEPA; July 2002. These supersede all previous qualifiers ever employed by BRL.</u>



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# Sample Information

Sample	Lab ID	Report Matrix	Type	Sampled	Received
BioReactor 1 Inf	1146034-01	FGD Wastewater	Sample	11/09/2011	11/11/2011
Hg Blk BioReactor 1 Inf	1146034-02	DIW	Field Blank	11/09/2011	11/11/2011
BioReactor 2 Inf	1146034-03	FGD Wastewater	QC Sample	11/09/2011	11/11/2011
Hg Blk BioReactor 2 Inf	1146034-04	DIW	Field Blank	11/09/2011	11/11/2011
BioReactor 2 Eff	1146034-05	FGD Wastewater	QC Sample	11/09/2011	11/11/2011
Hg Blk BioReactor 2 Eff	1146034-06	DIW	Field Blank	11/09/2011	11/11/2011

# **Batch Summary**

Analyte	Lab Matrix	Method	Prepared	Analyzed	Batch	Sequence
Hg	Water	EPA 1631	11/17/2011	11/18/2011	B111855	1100817



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# Sample Results

Sample	Analyte	Report Matrix	Fraction	Result	Qualifier	MDL	MRL	Unit	Batch	Sequence
<b>BioReactor 1 In</b> 1146034-01	<b>f</b> Hg	FGD Wastewater	Т	158		1.52	4.04	ng/L	B111855	1100817
<b>BioReactor 2 E</b> 1146034-05	<b>ff</b> Hg	FGD Wastewater	Т	86.8		1.52	4.04	ng/L	B111855	1100817
<b>BioReactor 2 In</b> 1146034-03	<b>f</b> Hg	FGD Wastewater	Т	152		1.52	4.04	ng/L	B111855	1100817
<b>Hg Blk BioRead</b> 1146034-02	c <b>tor 1 Inf</b> Hg	DIW	Т	0.15	U	0.15	0.40	ng/L	B111855	1100817
<b>Hg Blk BioRead</b> 1146034-06	ctor 2 Eff Hg	DIW	Т	0.15	U	0.15	0.40	ng/L	B111855	1100817
<b>Hg Blk BioRead</b> 1146034-04	ctor 2 Inf Hg	DIW	Т	0.15	U	0.15	0.41	ng/L	B111855	1100817



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# Accuracy & Precision Summary

Batch: B111855 Lab Matrix: Water Method: EPA 1631

Sample B111855-SRM1	Analyte Certified Reference Materia Hg	Native al (1145032,	<b>Spike NIST 1641d 1</b> 15.68	Result 000x dilution 15.46	Units on) ng/L	99% 85-115	RPD & Limits
B111855-MS4	<b>Matrix Spike (1146034-03)</b> Hg	152.2	606.1	879.9	ng/L	120% 71-125	
B111855-MSD4	Matrix Spike Duplicate (114 Hg	<b>16034-03)</b> 152.2	606.1	846.4	ng/L	115% 71-125	4% 24
B111855-MS3	<b>Matrix Spike (1146034-05)</b> Hg	86.76	404.0	518.9	ng/L	107% 71-125	
B111855-MSD3	Matrix Spike Duplicate (114 Hg	<b>16034-05)</b> 86.76	404.0	525.5	ng/L	109% 71-125	1% 24



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# Method Blanks & Reporting Limits

Batch: B111855 Matrix: Water Method: EPA 1631

Analyte: Hg

Sample	Result	Units
B111855-BLK1	0.09	ng/L
B111855-BLK2	0.05	ng/L
B111855-BLK3	0.09	ng/L
B111855-BLK4	0.07	na/L

 Average: 0.08
 Standard Deviation: 0.02
 MDL: 0.15

 Limit: 0.50
 Limit: 0.10
 MRL: 0.40



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## **Instrument Calibration**

**Sequence:** 1100817 **Total Mercury and Mercury Speciation by CVAFS** Instrument: THG-10

Method: EPA 1631

Date: 11/18/2011 Analyte: Hg

Lab ID	True Value	Result	Units	REC	C & Limits
1100817-IBL1		4.33	pg of Hg		
1100817-IBL2		6.20	pg of Hg		
1100817-IBL3		5.46	pg of Hg		
1100817-IBL4		5.40	pg of Hg		
1100817-CAL1	25.00	25.61	pg of Hg	102%	
1100817-CAL2	100.0	103.0	pg of Hg	103%	
1100817-CAL3	500.0	477.3	pg of Hg	95%	
1100817-CAL4	2500	2539	pg of Hg	102%	
1100817-CAL5	10000	9801	pg of Hg	98%	
1100817-ICV1	1568	1546	pg of Hg	99%	85-115
1100817-CCB1		8.48	pg of Hg		
1100817-CCV1	500.0	493.6	pg of Hg	99%	77-123
1100817-CCV2	500.0	552.6	pg of Hg	111%	77-123
1100817-CCV3	500.0	505.6	pg of Hg	101%	77-123
1100817-CCV4	500.0	445.5	pg of Hg	89%	77-123
1100817-CCV5	500.0	524.3	pg of Hg	105%	77-123



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# **Sample Containers**

	<b>ID:</b> 1146034-01 <b>ple:</b> BioReactor 1 Inf		•	rt Matrix: FGD Wastewater ble Type: Sample		Collected: 11/09/2011 Received: 11/11/2011					
Des A	Container Bottle FLPE Hg-T	Size 250mL	<b>Lot</b> 71470160 10	рН	Ship. Cont. Cardboard Box						
	ID: 1146034-02 ple: Hg Blk BioReactor 1 Inf		•	rt Matrix: DIW le Type: Field Blank			ted: 11/09/2011 ved: 11/11/2011				
Des A	Container Bottle FLPE Hg-T	Size 250mL	<b>Lot</b> 71470160 10	Preservation none	P-Lot n/a	рН	Ship. Cont. Cardboard Box				
	ID: 1146034-03 ple: BioReactor 2 Inf		•	rt Matrix: FGD Wastewater			cted: 11/09/2011 ved: 11/11/2011				
Des A	Container Bottle FLPE Hg-T	Size 250mL	Lot Preservation P-Lot 71470160 none n/a 10		рН	Ship. Cont. Cardboard Box					
	ID: 1146034-04 ple: Hg Blk BioReactor 2 Inf		•	rt Matrix: DIW le Type: Field Blank		Collected: 11/09/20 Received: 11/11/20					
Des A	Container Bottle FLPE Hg-T	Size 250mL	<b>Lot</b> 71470160 10	Preservation none	P-Lot n/a	рН	Ship. Cont. Cardboard Box				
	ID: 1146034-05 ple: BioReactor 2 Eff		•	rt Matrix: FGD Wastewater			cted: 11/09/2011 ved: 11/11/2011				
<b>Des</b> A	Container Bottle FLPE Hg-T	Size 500mL	Lot 71490150 70	Preservation none	P-Lot n/a	рН	Ship. Cont. Cardboard Box				
	ID: 1146034-06 ple: Hg Blk BioReactor 2 Eff		-	rt Matrix: DIW le Type: Field Blank		Collected: 11/09/201 Received: 11/11/201					
Des A	Container Bottle FLPE Hg-T	Size 250mL	Lot 71470160 10	Preservation none	P-Lot n/a	рН	Ship. Cont. Cardboard Box				



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# **Shipping Containers**

### **Cardboard Box**

**Received:** November 11, 2011 9:00 **Tracking No:** 4726 7966 5796 via FedEx

Coolant Type: None Temperature: ambient

Description: Cardboard Box Damaged in transit? No Returned to client? No Custody seals present? No Custody seals intact? No COC present? Yes

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*thomas.d.johnson@siemens.com	12)Seal/Lock Opened By	10) Seal/Lock Opened By	8)Accepted By:	6)Accepted By:	4) Accepted By	2) Accepted By					t-	725	1	1 1320		1/41/1 /20			Date Time	Sampling conducted: 2n	Customer to comple appropriate non-shade	MR.		Brooks Rand	Logged By	ORDER#	CORD AND A
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C. SHARMA (980) 875-52

DUKE ENERGY 13339 HAGERS FERRY RD BLDG # 7405 HUNTERSVILLE, NC 28078 SHIP DATE: 10NOV11 ACTWGT: 5.6 LB CAD: 798987/CAFE2509 DIMS: 11×11×11 IN BILL SENDER

™ATTN: MICHELLE BRISCOE BROOKS RAND 3958 6TH AVENUE NW

SEATTLE WA 98107

(206) 632-6206

REF

Fedex

| The state of the state

TRK# 4726 7966 5769

FRI - 11 NOV A1 PRIORITY OVERNIGHT

NC BFIA

98107 WA-US SEA



Bokeach 2 ETF

714901 5076

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CHAIN OF CUSTODY RECORD AND ANALYSIS REQUEST FORM Page 28 of 29 **Analytical Laboratory Use Only Duke Energy Analytical Laboratory** Page 1 of 2 Duke Energy<sub>s</sub> MATRIX: OTHER ORDER# DISTRIBUTION Mail Code MGO3A2 (Building 7405) Originating 13339 Hagers Ferry Rd ORIGINAL to LAB Huntersville, N. C. 28078 **COPY to CLIENT** SAMPLE PROGRAM Ground (704) 875-5245 NPDES Fax: (704) 875-4349 **Drinking Water** UST Belews - FGD RCRA Waste Cooler Temp (C) WWTS Bi-Monthly Sampling) AS&C 15 Preserv.:1=HCL 4)Fax No: 2) Client: Bill Kennedy, Melonie Martin, PO#133241 2=H2SO4 3=HNO3 Wayne Chapman, Tom Johnson \*\* 4=Ice 5=None 6)Process: speciation - vendor to AS&C (Important to place filled bottle back into both baggies) 5)Business Unit: Required 16Analyse Mail Code: dig.) 10)Reso. Center: 9)Res. Type: 8)Oper. Unit: Customer to complete all soluble (no appropriate non-shaded areas. Br (Dionex) Hg - 245.1 Sampling conducted: 2nd and 4th Wednesday Metals\* LAB USE ONLY 18 Grab Se Speciation Bottle TDS Se, Se, <sup>13</sup>Sample Description or ID Date Time Signature 11/9/11 7:30 w. workens **FGD Purge Eff** 11/9/11 7:35 1 EQ Tank Eff. 1 11/9/11 7:40 BioReactor 1 Inf 11/9/11/7:45 88 BioReactor 2 Inf 11/9/11 750 1 BioReactor 2 Eff 89 11/9/11 7155 Filter Blk 11/9/11/8:00 Metals Trip Blk Filtering of the Se is performed in the field please provide a filter blank too. Customer to sign & date below - fill out from left to rigi Date/Time 1) Relinquished By <sup>22</sup>Requested Turnaround 13:00 hrs turnaround. W. Workman Date/Time 14 Days r, IMPORTANT! 6)Accepted By: \*7 Days Date/Time 8)Accepted By: 7)Relinquished By Date/Time \*Other 10) Seal/Lock Opened By \* Add. Cost Will Apply 12)Seal/Lock Opened By Date/Time 1)Seal/Locked By Comments As, Cr, Cu, Ni, Se, Ag, Zn by IMS thomas.d.johnson@siemens.com

Digestions = TRM

\* B by ICP

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